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Reconstruction of anthropogenic activities in legacy sediments from the Eure River, a major tributary of the Seine Estuary

3 (France)

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29 Abstract

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31 Most rivers worldwide are contaminated by various trace metals from different origins, which may be stored for considerable periods of time in depositional areas. Most of these 32 studies are focused on the main river of a watershed and the tributaries are often neglected 33 34 which can be important sources of contamination. The aim of this study was to reconstruct the anthropogenic activities that occurred in the Eure River, tributary of the Seine Estuary, since 35 36 the 1940s using "legacy sediments". The results showed that the temporal trends of trace 37 metals were not related to detrital inputs and TOC variations but with the industrial history of the Eure River watershed. The high levels of Zn, Cu, and Ni during the 1950s and the 1960s 38 and the decrease with the decline of the probable main source of release showed the 39 watershed reactivity to anthropogenic activities. The high levels of Pb during the 1990s and 40 the 2000s showed that the watershed reacted immediately to anthropogenic pressures. The Pb 41 42 levels remained important after the cease of industrial activity, showing that a resilience period is necessary for the system, and that interactions between human activities and the 43 environment go beyond of the activities themselves. 44

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46 **1. Introduction**

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Many researchers agree that the Earth has entered a new geological period, the Anthropocene (Crutzen and Stoermer, 2000; Crutzen, 2006; Steffen et al., 2011; Lewis and Maslin, 2015). While the beginning of this period is the subject of intense debate (Lewis and Maslin, 2015), there is consensus that humans have seriously impacted their environment during this period (Steffen et al., 2011). Such human activities as agriculture, forestry, mining,

and water storage and diversion contribute significantly to landscape change and 53 environmental contamination. The sediments generated by the erosive actions of humans have 54 received increasing attention in recent years and have even been categorised as a particular 55 type, known as legacy sediments (James, 2013, 2018); these are generally defined as alluvial 56 57 sediments that are deposited as a result of anthropogenic disturbances within a watershed (James, 2013). Among the several sedimentation and storage zones throughout the watershed, 58 there is particular concern about how legacy sediments trapped behind dams are managed (i.e. 59 dredging). Structures such as dams change the flow of a river to achieve particular objectives 60 (e.g. energy production, flood control, and navigation) and serve to modify the hydrodynamic 61 cycle and thus the sediment storage in catchment (Nilsson et al., 2005; Yang et al., 2006). 62 63 Many studies have examined the storage of metallic and organic contaminants behind dams (e.g. Shuman, 1995; Müller et al., 2000; Bednarek, 2001; Stanley and Doyle, 2003; Audry et 64 al., 2004; Ashley et al., 2006; Devault et al., 2009; Dhivert et al., 2015; Evans, 2015). Hence, 65 legacy sediments can be viewed as the industrial and agricultural testimonies of past 66 anthropogenic activities in, and can reflect the trajectory of, any studied watersheds. In 67 addition, estuaries are dynamic environments where it is difficult to reconstruct temporal 68 trends of contamination and the origins of these latter from legacy sediments. So, determining 69 the origins of contamination in an estuary requires to study its tributaries. 70

The Seine River watershed (France) provides an example of a basin experiencing 71 anthropogenic activities. The industrial revolution increased the anthropogenic pressure on the 72 Seine River considerably over a period of about 150 years, resulting in specific and drastic 73 changes in the stream morphology. These modifications meant that the flow of the Seine 74 River was regulated, mainly to facilitate navigation in the nineteenth century (Horowitz et al., 75 1999; Lestel et al., 2019). The morphological modifications also impacted the tributaries of 76 the Seine River. Thus, the Eure River, the most important contributor to the Seine Estuary, 77 saw its outlet diverted 11 km downstream between 1929 and 1939. All these adjustments have 78

promoted the establishment of depositional environments favouring the storage of legacy 79 sediments suitable for reconstructing anthropogenic activities in the watershed. However, if 80 the history of the Seine River is well known, there is less historical information about its 81 tributaries; the latter could potentially be the main source of contamination in the lower 82 reaches of the watershed, i.e., the estuary (e.g. the Gironde Estuary; Audry et al., 2004; 83 Masson et al., 2006). Most sediment cores covering at least the last 70 years were extracted 84 along the Seine Estuary (Van Metre et al., 2008; Vrel, 2012; Vrel et al., 2013; Kaci et al., 85 2014, 2016) and along the fluvial part of the Seine River, upstream from the Poses Dam 86 (Figure 1A; Bonté et al., 2004; Meybeck et al., 2007; Ayrault et al., 2010a; Le Cloarec et al., 87 2011; Tamtam et al., 2011; Avrault et al., 2012; Vrel et al., 2013; Lorgeoux et al., 2016). In 88 89 contrast, there are few sediment records for the tributaries (Figure 1B; Avrault et al., 2010b; Le Cloarec et al., 2011; Ayrault et al., 2012). Unfortunately, the geographic locations of the 90 cores from the Oise and Marne rivers restricted the reconstruction of the sedimentary and 91 contamination history to a limited area of these two tributaries. The best location for coring is 92 at the outlet of a tributary, where the geochemical signatures of the river being studied can 93 best be captured. 94

This is the reason that anthropogenic activities in the Eure River watershed were 95 investigated, for the first time, using sediment cores collected from two ponds near the outlet. 96 As recently raised by Bábek et al. (2020), the spatial distribution of sediments in the 97 depositional zones (behind a dam in the case of this study) and thus the spatial distribution of 98 sediment-associated contaminants, is rarely studied. In addition, it is generally accepted that 99 100 the studied sediment core is representative of the watershed signals, but without the absolute certainty that a local disruption could have disturbed the global recorded signal. Hence, it is 101 difficult to know if the studied sediment core is representative of (i) the whole depositional 102 zones and, (ii) the global signature of the inputs from the whole watershed. To circumvent this 103 uncertainty, sediment cores were collected in our study in two ponds in order to ensure that 104

the recorded sedimentological and geochemical signatures are not disturbed by local inputs 105 106 and can be considered as representative of the whole watershed. In addition, and for each ponds, the representativeness of the sediment cores was investigated with ground-penetrating 107 radar profiles. Our study also proposes an original coupling between sedimentological and 108 109 geochemical analyses to assess the deposition conditions and the nature of the sediment deposits as they can influence the temporal trends of trace metal elements TME (Dhivert et 110 al., 2015). Lastly, the morphological changes modifying the depositional conditions have been 111 112 studied by using historical documentaries as seen in previous studies (Pittam et al., 2009; Grygar et al., 2016). 113

Hence, combining a comprehensive set of geophysical, sedimentological, geochemical 114 115 methods and, historical documentaries, our main objectives were to (i) determine the consequences of the morphological modifications of depositional zones during the diversion 116 of the Eure River outlet on the sedimentary dynamic, (ii) determine the nature of these legacy 117 sediment deposits and (iii) reconstruct the temporal trends of anthropogenic activities with a 118 focus on TME considered as markers of industrial activities in the Eure River watershed. This 119 120 study also discussed the reactivity of the watershed to anthropogenic activities, and the resilience of the system to overcome these stresses. 121

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123 **2. Material and Methods**

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125 2.1. Setting

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127 The Seine River flows into the Paris Basin. Sedimentary formations (Jurassic limestone and 128 marls, Cretaceous chalk, carbonaceous alluvial, and Tertiary quartz deposits) cover most of 129 the basin, with carbonate formations dominating (Thibert, 1994). The Eure River watershed (Figure 1B) is underlain by a chalk formation from the Upper Cretaceous, covered by sandyclay deposits from the Tertiary and Quaternary deposits (Quesnel, 1997; Laignel et al., 1998). The Eure River is the fourth largest tributary of the Seine Basin in terms of area (6 017 km²), and the fifth in terms of flow at the outlet ($Q_{mean} = 22.1 \pm 6.7 \text{ m}^3 \text{ s}^{-1}$) for the period from 1971 to 2019 (www.hydro.eaufrance.fr).

Until the end of the nineteenth century, there were numerous islands spread through the channel of the Seine Estuary. At that time, the Eure River outlet was more than 11 km upstream of the present outlet (Figure 2A). Therefore, the studied coring sites at Martot and Les Damps did not exist (see section 2.2) and were in the left branch of the Seine Estuary, which was a dynamic riverine environment, unfavourable to sediment deposition.

140 Between 1861 and 1864, the Martot Dam (needle dam), the first of its scale in the Seine Estuary, was built in two parts (169.6 m + 135.0 m) across two branches of the Seine River 141 (Figure 2B). During the 1880s, the Poses Dam was constructed further upstream (Figure 1A). 142 These two dams were built in a section of the Seine Estuary where navigation was made 143 difficult by the presence of the islands and natural weirs. The first major modification that 144 impacted the Eure River dates back to the 1930s (1929–1935), when the Eure River outlet 145 was moved 2 km downstream by following a former part of the left branch of the Seine River 146 (Figure 2C). This morphological modification formed a pond connected to the Eure River 147 (Les Damps Pond). The entire section was completely disrupted during the 1930s, and the 148 channel of the Seine River was deeply restructured. During this period, the channel 149 morphology of the Seine River was modified, in some cases by passing through former 150 151 islands or by filling connections between the many islands present in its channel. Between 1937 and 1939, the Eure River outlet was moved 9 km downstream by following the former 152 branch of the Seine River and was relocated at the Martot Dam. At the same time, a dike was 153 built in the middle of the Martot Dam in the direction of the flow to isolate a pond (Martot 154 Pond). In 1939, the connection between Martot Pond and the Seine River was filled to 155

calibrate the Seine River channel while a connection between Martot Pond and the Eure River
was opened thereafter. The Martot Dam became obsolete when the connections between the
islands were filled and was partially removed between 1938 and 1941, leaving only the part
across the branch of the Eure River (Figure 2D).

160 The area did not experience further evolution until 2017 when the Martot Dam was removed 161 following the Water Framework Directive (2000/60/CE), to facilitate the continuity of 162 sediment transport.

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164 2.2. Sampling sites

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166 2.2.1. Upstream site: Les Damps Pond

Les Damps Pond (formed between 1929–1935) is upstream of the Eure River spillway (Figure 3A). The pond, which has an average depth of 50 cm and an area of less than 1 ha, is connected to the Eure River, even during low water periods, and continually accumulates suspended particulate matter (SPM) from the Eure River watershed (Figure 3B). Because the site is 2 km upstream of a spillway, the tides have no noticeable effect, even in the case of a high tidal coefficient.

The cores were extracted from this pond in January 2015 and 2017, using a gravity corer (UWITEC) and PVC tubes with a diameter of 90 mm. The water-sediment interface was preserved during coring (Table 1).

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177 2.2.2. Downstream site: Martot Pond

Cores were also extracted from Martot Pond (area ~ 7 ha), in the lower reaches of the study area (Figure 3A). Martot Pond is affected by the waters of the Seine River during tidal flows through the Eure River outlet. However, the impact of the tide is limited and temporally variable, as its flow persists for about 3 h and does not occur every day, because the dam, located 200 m downstream from the connection with this pond, stops tidal flows for tidal coefficients lower than 70 (until the removal of the dam in 2017) (Figure 3C). Thus, to ensure Martot Pond was not impacted by the Seine River flow, results from this core site were compared with those from Les Damps Pond. The Martot Dam also regulated the water level in the river and the latter was continuously connected to the Martot Pond, even during periods of low flow.

The cores were extracted from this pond in January and May 2015 and February 2017, using a gravity corer (UWITEC) and PVC tubes with a diameter of 90 mm. The water-sediment interface was preserved during coring (Table 1).

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192 2.3. Geophysical investigation: GPR

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Ground penetrating radar (GPR) is a non-invasive geophysical technique that detects 194 electrical discontinuities. It is based on the generation, transmission, propagation, reflection, 195 and reception of discrete pulses of high frequency electromagnetic energy (MHz). For several 196 years, it has been particularly used in the study of sedimentary structures and deposition 197 environments in fluvial environments (Huggenberger et al., 1994; Asprion and Aigner, 1999; 198 Beres et al., 1999; Bristow and Jol, 2003; Neal, 2004; Kostic et al., 2005; Huggenberger and 199 Regli, 2006; Bábek et al., 2008; Słowik, 2015; Gu et al., 2019). In this study, the surveys were 200 carried out in an aquatic environment (Water GPR-WGPR) because it is possible to tow the 201 202 antennas of the geological radar in or behind a boat (Mellett, 1995; Lin et al., 2009; Sebok et 203 al., 2018). The GSSI SIR 4000 acquisition system (Geophysical Survey System Inc., Salem, USA) was connected to a Trimble GPS with a 400 MHz antenna. The instrumentation was 204 loaded on a high density polyethylene boat. A total of five longitudinal profiles was acquired 205 206 closest to the sampled cores, at both of the two sites (Figures 3B & 3C). The data were processed with Radan 7 software (GSSI copyright) to obtain interpretable radargrams. A 207

208	velocity of 0.075 m ns ⁻¹ was determined using the hyperbola method and the correlation with
209	sediment cores. This is of the same order of magnitude as the measurements made by Lin et
210	al. (2009) (0.076 m ns ⁻¹) and O'Driscoll et al. (2010) (0.06 m ns ⁻¹) on comparable sediments.

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212 2.4. Sedimentological analysis

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214 2.4.1. Grain size distribution

215 The grain size distribution can influence the behaviour of chemical compounds. Radioactive tracers (²¹⁰Pb, ¹³⁷Cs, and ²⁴¹Am) are preferentially adsorbed onto fine particles (Livens and 216 Baxter, 1988; Walling and Woodward, 1992; Wang and Cornett, 1993; He and Walling, 1996; 217 218 Monna et al., 1998; Gil-García et al., 2009) as well as organic and metallic contaminants (Horowitz and Elrick, 1987; Walling et al., 2003; Grosbois et al., 2006; Devault et al., 2009; 219 Coynel et al., 2016). Thus, the grain size distribution can have a considerable impact on 220 221 radioactive tracers and TME concentrations along sediment cores (He and Walling, 1996). Differences in grain size are also excellent indicators of flood events or anthropogenic 222 223 activities (e.g. changes in land use) (Kurashige and Fusejima, 1997; Weltje, 2012; Toonen et al., 2015; Collins et al., 2017). 224

The grain size distribution of the sediment cores was measured by laser diffraction (LS 13320 Particle Size Analyser Beckman CoulterTM). The measurements were realised every cm by integrating 1 cm on the DAM15-02 and MAR15-01 cores.

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229 2.4.2. Total organic carbon content

The total organic carbon (TOC) and organic matter (OM) quality can also influence TME concentrations (Audry et al., 2006; Tseng et al., 2001; Masson et al., 2011; Petit et al., 2013; Coynel et al., 2016). Moreover, variations in TOC and related parameters (e.g. HI and OI) provide information on the hydrological dynamics and anthropogenic impact in a watershed.

The TOC content was measured using Rock-Eval 6 (RE6) pyrolysis ("Turbo" model RE6 234 pyrolyzer, at the ISTO laboratory, University of Orléans), as described in Copard et al. 235 (2006). The OM quality was obtained from measuring HI (hydrogen index, in mg HC g⁻¹ 236 TOC) and OI (oxygen index, in mg O_2 g⁻¹ TOC). HI corresponds to the quantity of HC 237 released relative to TOC, while OI reflects the quantity of oxygen released as CO and CO2 238 relative to TOC. Used together, these two parameters in the pseudo Van-Krevelen diagram 239 (HI vs OI) can support the identification of the OM origin and its subsequent processes 240 (Tissot and Welte, 1984). These variables were measured on the DAM17-02 (at intervals of 2 241 cm) and MAR15-01 cores (at intervals of 1 cm). 242

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244 2.4.3. Magnetic susceptibility

The magnetic properties of accumulated stored sediments are generally used to identify detrital inputs (e.g. from floods) (Debret et al., 2010) and anthropogenic modifications (e.g. metallic pollution in soils and sediments) (Hanesch and Scholger, 2002; Knab et al., 2006; Magiera et al., 2006; Blaha et al., 2008). The magnetic properties of the sediment can also be used to determine the origin of the particles (Caitcheon, 1998; Hatfield and Maher, 2008, 2009; Hatfield and Stoner, 2013; Collins et al., 2017).

The magnetic susceptibility (MS) of the cored sediments was measured using a Bartington MS2E point sensor as described in Debret et al. (2010). The MS of the sediment from the DAM15-02, DAM15-03, DAM17-02, MAR15-01, and MAR15-05 cores was determined.

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255 2.4.4. Spectrocolorimetry

Various parameters that can be determined using a spectrocolorimeter, such as reflectance (L*) and the first derivative reflectance spectra (FDS), may be used to determine the nature and the origin of sediment deposits, and sediment dynamics (Debret et al., 2006, 2011; Sebag et al., 2013). L* describes the lightness of the sediment without any information on the nature

260	of the sediment. FDS provides qualitative information about the major components (such as
261	clay and carbonates) of the sediment deposits. The Q7/4 diagram (700 nm/400 nm ratio vs
262	L*) proposed by Debret et al. (2011) is used to determine the sedimentary dynamics.

A Konica Minolta CM 2600d was used to measure the intensity of the reflectance of the sediment in the visible domain (360–740 nm with a spacing of 10 nm) as described in Debret et al. (2011).

These measurements were obtained for the DAM15-02, DAM15-03, DAM17-02, MAR1501, MAR15-02, MAR15-03, MAR15-04, MAR15-05, and MAR16-02 cores.

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269 2.5. Geochemical analysis by XRF core scanning (XRF-CS)

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X-Ray fluorescence spectrometry (XRF) is used to determine the major and trace element
composition of sediment deposits (Richter et al., 2006). The XRF core scanning data can be
used to describe particular TME contamination in sediment cores (Battiston et al., 1989;
Dickinson et al., 1996; Lepland et al., 2010; Sabatier et al., 2014; Guédron et al., 2016;
Hennekam et al., 2019). The data presented were normalised by Ti (considered as a better
"normalising element" than Al) to remove possible variations because of detrital inputs
(Kylander et al., 2011; Duan et al., 2014; Bábek et al., 2015; Chawchai et al., 2016).

The relative contents of the major and trace elements were measured using an XRF Avaatech core scanner (EDYTEM, University of Savoie Mont Blanc). An X-Ray beam was generated with a rhodium anode and a 125 µm beryllium window, which allows a voltage range from 7 to 50 kV and a current range of 0 to 2 mA. The analytical settings were adjusted at 10 kV and 1 mA to detect light elements, and trace elements (namely Pb, Cu, Zn, and Ni) were detected in a second run, performed at 30 kV for 0.75 mA. Each individual power spectrum was transformed by deconvolution into its relative contents (intensities), expressed in counts per second (cps). Measurements were realised every 5 mm for the MAR15-01 and
DAM17-02 cores and every 2 mm for the MAR16-02 and DAM15-02 cores.

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288 2.6. TME analysis

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Microwave-assisted sediment digestion for particulate trace metal analysis (Pb, Zn, Cu, 290 presented in this study) was performed on samples collected in the MAR16-02 core (0.5 g of 291 dry, powdered and homogenised sediment) with aqua regia (HNO3: 3 mL TraceMetalTM 292 Grade; HCl: 9 mL TraceMetalTM Grade). Following the mineralisation, the solutions were 293 diluted in 100 ml of Milli-Q[®] water. Particulate trace metal concentrations were measured by 294 inductively-coupled-plasma atomic emission spectroscopy (ICP-AES) (iCAP 6000 Series, 295 Thermo Fischer®) performed with external calibration. The accuracy and precision of the 296 measurements were checked against a certified reference material (Trace Element on Fresh 297 Water Sediment CNS301-04-050, Sigma Aldrich) and the results showed accuracies of ≥ 98 298 % with a precision of ≤ 4 % (RSD). All of the analyses were carried out in triplicate (standard 299 300 deviation ≤ 1.1 %).

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- 302 2.7. Short-lived radionuclides
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The activities of ²¹⁰Pb in excess (²¹⁰Pb_{ex}), ¹³⁷Cs, and ²⁴¹Am were determined on samples from the MAR15-01 core (at steps of 1, 4, or 6 cm) using a germanium spectrometer with ultra-low background noise at the Laboratoire Souterrain de Modane (LSM). The age model was computed with the *serac* R package (https://github.com/rosalieb/serac, (Bruel and Sabatier, Submitted).

309

310 **3. Results**

312 3.1. Sedimentological characterisation of the sediment cores

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314 *3.1.1.* Les Damps Pond

The sediment cores all showed similar sedimentary facies called U1 (Figures 4A1 & 4A2). The grain size distribution was homogeneous (median (D_{50}) = 29.5 ± 6.7 µm) and showed only one population, centred around the D_{50} . (Figure 4B). The TOC content was high and constant (TOC_{mean} = 5.36 ± 0.47 %). HI and OI were also constant, and the values of HI were higher than those of OI (HI_{mean} = 302 ± 23 mg HC g⁻¹ TOC and OI_{mean} = 145 ± 10 mg O₂ g⁻¹ TOC) (Figure 4C).

The MS values were high and constant between 60 and 90 cm (MS_{mean} = 12 ± 2) but had a peak at 50–60 cm. The values decreased between 50 and 30 cm and were constant above 30 cm (MS_{mean} = 6 ± 1) (Figure 4D).

The values of L* were low and constant (L*_{mean} = $28.5 \pm 2.3 \%$), with values between 22 and 38 % (Figure 4D).

The intensities of FDS were high at wavelengths between 675 and 735 nm. This signature was continuous, and the intensity did not vary, which may mean that the origin of the sedimentary inputs remained the same throughout the sedimentation (Figure 4E).

The Pb/Ti ratio was low and constant (Pb/Ti_{mean} = 0.64 ± 0.12) between 30 and 80 cm but increased at 30 cm. The values of the ratio were generally high between 0 and 30 cm (Pb/Ti_{mean} = 2.26 ± 0.58) and decreased from 15 cm to the surface (Figure 4F). The Zn/Ti, Cu/Ti, and Ni/Ti ratios showed the same trend. These three ratios were high between 30 and 80 cm and reached maximum values between 50 and 60 cm (Zn/Ti_{mean} = 2.04 ± 0.24 , Cu/Ti_{mean} = 1.43 ± 0.71 , and Ni/Ti_{mean} = 0.04 ± 0.01). Around 30 cm, the ratios decreased and were constant between 0 and 30 cm (Figure 4F). 337 *3.1.2. Martot Pond*

The sediment cores showed two sedimentary facies delimited by a transition zone at 75–80 cm. In this section, the results from the MAR15-01 core are presented. The upper facies (0–75 cm) is referred to as U1, while the lower facies (75–138 cm) is referred to as U2 (Figure 5A).

341 • U2 facies:

The grain size distribution was homogeneous (median (D_{50}) = 13.8 ± 7.0 µm) and showed one population centred around D_{50} (Figure 5B). The TOC content was low and constant (TOC_{mean} = 1.75 ± 0.33 %). HI was constant (HI_{mean} = 147 ± 24 mg HC g⁻¹ TOC) and lower than OI (OI_{mean} = 260 ± 35 mg O₂ g⁻¹ TOC). OI increased, peaked at 103.5 cm (OI = 347 mg O₂ g⁻¹ TOC), then decreased. The only exception to this trend was at the depth of 128.5 cm (Figure 5C).

The MS was low and L* was high and constant ($L_{mean}^* = 44.4 \pm 2.1 \%$), with values between 40 and 50 % (Figure 5D).

The intensities of FDS were high for wavelengths between 365 and 550 nm. A second signature, less intense, was also visible for high wavelengths (675–735 nm). This indicates that several signatures were marked within the facies and the small variations in intensity suggest the origin of the whole U2 facies was unique (Figure 4E).

The Pb/Ti, Zn/Ti, Cu/Ti, and Ni/Ti ratios were low and constant (Pb/Ti_{mean} = 0.33 ± 0.10 ,

355 $Zn/Ti_{mean} = 0.54 \pm 0.11$, Cu/Ti_{mean} = 0.18 ± 0.05, and Ni/Ti_{mean} = 0.04 ± 0.01) and were higher

- only at the transition between the two facies in the 75–80 cm depth interval (Figure 5F).
- **357** U1 facies:

The grain size distribution was homogeneous (median (D_{50}) = 33.9 ± 7.8 µm) and had one population centred around D_{50} (Figure 5B) as in Les Damps Pond. The TOC content was twice as high in U1 (TOC_{mean} = 3.88 ± 1.49 %) as in U2, and the values were particularly high near the top of the core (TOC = 8.26 ± 0.19 % between 9 and 12 cm). HI increased upwards from the transition between U1 and U2 and stabilised at a depth of 66.5 cm. OI continued to decrease in U1 and stabilised at a depth of 66.5 cm in U1. At the opposite end of the U2 facies, HI was high (HI_{mean} = 279 ± 23 mg HC g⁻¹ TOC) and OI was low (OI_{mean} = 150 ± 17 mg O₂ g⁻¹ TOC) (Figure 5C) as described along the U1 facies in Les Damps Pond.

The U1/U2 transition was marked by a strong increase in MS and a decrease in L*. The MS values were high ($MS_{mean} = 25 \pm 7$) from 45 to 75 cm but were lower ($MS_{mean} = 8 \pm 4$) between 0 and 45 cm (Figure 5D). The L* was lower in the U1 facies ($L*_{mean.} = 34.1 \pm 4.2 \%$) than in the U2 facies, and was almost the same as the values between 24 and 40, and decreased next to the top of the core (Figure 5D). The sediment colour was similar to that in the U1 facies in Les Damps Pond.

The intensities of FDS were high at high wavelengths (675–735 nm), but, unlike the U2 facies, low wavelength signatures were not noticeable. This indicates a major change in the nature of the sediment stored in Martot Pond (Figure 5E). Moreover, the signature was continuous and the intensity did not vary as in Les Damps Pond.

The Pb/Ti ratio increased at the U1/U2 transition but remained stable to a depth of 35 cm 376 (Pb/Ti_{mean} = 1.06 ± 0.15 between 35 and 75 cm). The ratio was higher from 35 to 10 cm 377 (Pb/Ti_{mean} = 3.17 ± 0.53 between 10 and 35 cm) and trended to decrease closer to the top of 378 the core (Figure 5F). The patterns in the Zn/Ti, Cu/Ti, and Ni/Ti ratios were similar. Indeed, 379 from the transition between U1 and U2, the ratios increased and peaked between 50 and 60 380 cm (Zn/Ti_{mean} = 4.79 ± 0.73 , Cu/Ti_{mean} = 1.19 ± 0.20 , and Ni/Ti_{mean} = 0.11 ± 0.01). The ratios 381 decreased from 50 to 35 cm and then stabilised above 35 cm (Figure 5F). The variations in the 382 Pb/Ti, Zn/Ti, Cu/Ti, and Ni/Ti ratios generally followed a similar pattern to those described 383 for Les Damps Pond. 384

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387

The trends in the Pb/Ti, Zn/Ti, and Cu/Ti ratios for the top 77 cm of the MAR16-02 core and the U1 facies of the MAR15-01 core were similar, which shows that this facies was also well represented within MAR16-02. The Pb, Zn, and Cu concentrations were measured at the depths for which the XRF ratios reached maximum and minimum values within the MAR16-02 core (Figure 6).

The Pb concentration was constant to a depth of 35 cm ($Pb_{mean} = 120 \pm 28 \text{ mg kg}^{-1}$), apart 393 from at 65 cm (Pb = 190 mg kg⁻¹). The Pb concentrations increased in the same way as the 394 Pb/Ti ratio, and the concentrations were highest at 12 cm (Pb = 669 mg kg⁻¹). Near the top of 395 the core (4 cm), the Pb concentration decreased, similar to the Pb/Ti ratio, and at 299 mg kg⁻¹ 396 397 it was less than half the maximum concentration at 12 cm but almost 2.5 times the average concentration below 35 cm (Figure 6). The Zn and Cu concentrations were 415 mg kg⁻¹ and 398 113 mg kg⁻¹ at 77 cm, respectively. The concentrations of these two TMEs peaked at 65 cm 399 $(Zn = 905 \text{ mg kg}^{-1}, Cu = 399 \text{ mg kg}^{-1})$. Between 10 and 40 cm, the Zn and Cu concentrations 400 were less than half and less than a quarter of the maximum concentrations, respectively, and 401 the Zn/Ti and Cu/Ti ratios were also low close to the top of the MAR16-02 core (Figure 6). 402 The Zn and Cu concentrations at 4 cm were lower than those measured at 77 cm. 403

404

405 3.3. Age model of the MAR15-01 core

406

Recent deposit dating and sedimentation rate estimates have been realised from short-life
radionuclides (Robbins and Edgington, 1975; Appleby and Oldfield, 1978; Appleby et al.,
1991; He and Walling, 1996; Mabit et al., 2014). The sedimentation rate was determined from
the ²¹⁰Pb_{ex} by subtracting the ²²⁶Ra activity from the ²¹⁰Pb_{total} (Krishnaswamy et al., 1971).
The ¹³⁷Cs and ²⁴¹Am activities were used to identify nuclear weapon testing with maximum

activity in 1963, and the Chernobyl accident in 1986 and are used to confirm the age model
established with ¹⁰Pb_{ex}.

The activity of ²¹⁰Pb_{ex} was measured to 80 cm and decreased almost continuously with 414 depth. A sedimentation rate of 12.73 mm y^{-1} (R² = 0.60) was estimated using the 'constant 415 flux, constant sedimentation rate' (CFCS) model (Krishnaswamy et al., 1971) (Figure 7A). 416 The ¹³⁷Cs and ²⁴¹Am activities peaked at 60 and 58 cm, respectively, corresponding to the 417 maximum fallout from atmospheric nuclear weapon testing in 1963 (Robbins and Edgington, 418 1975; Appleby et al., 1991) (Figure 7B). There was also a peak in the ¹³⁷Cs activity at 40 cm, 419 perhaps related to the Chernobyl accident in 1986 (Figure 7B). An age model was realised for 420 the U1 facies of the MAR15-01 core using the sedimentation rate obtained from the ¹⁰Pbex 421 422 (Figure 7C).

423

424 **4. Discussion**

425

426 4.1. Sedimentary filling in the depositional zones

427

As the sedimentological parameters are presented for one core from Martot Pond (two cores 428 in the case of Les Damps Pond), it is essential to ensure that the studied cores are 429 representative of sediment records in the entire respective pond. Concerning Martot Pond, a 430 sediment thickness between 2.0-2.5 m is showed by the radargram GPR_MAR_08 (Figure 431 8A). The reflector R1 made it possible to distinguish two islands without a well-differentiated 432 433 structure on this scale, which may correspond to parts of the central island that separated the two branches of the Seine River (Figure 2). A second reflector (R2, solid red line) between 434 350 m and 550 m separates U1 and U2 and this interpretation is justified by the MAR15-02 435 core in which this limit is observed at 85 cm (corresponding to the transition between U1 and 436 U2 observed on MAR15-01 core at 75-80 cm). Beyond this section, it is more difficult to 437

locate R2, but the position of the MAR15-03, MAR15-04 and MAR15-06 cores, with the aid
of some clues, allowed us to position R2 (red dotted line). Geophysical prospecting shows a
homogeneous filling within Martot Pond and thus validates the representativeness of the
studied core.

442 Concerning Les Damps Pond, two quality profiles have been obtained (Figures 8B & 8C) in which a reflector (R3) showed the lower limit of U1. In this facies, few continuous structures 443 were detectable, probably because of the fine accumulated sediments and the monotony of the 444 sediment as observed in the DAM15-02 and DAM17-02 cores. However, under R3, it was 445 possible to distinguish less powerful reflectors, probably attributed to the presence of a bed of 446 coarser material (Figure 8B). This reflector was therefore interpreted as the upper limit of the 447 former stream-bed of the Seine River. Hence, the two studied sediment cores are 448 representative of the sedimentary records of the entire pond. 449

450

451 4.2. Identification of the deposition conditions and sediment origin

452

453 The grain size in the U2 facies ($D_{50} = 13.8 \pm 7.0 \mu m$) of Martot Pond and in the U1 facies of Martot and Les Damps Ponds ($D_{50} \sim 30 \mu m$) suggests a weak sedimentation dynamic, that is, 454 deposition under very low flow conditions, but also suggests two distinct unidentified origins. 455 U2 was also characterised by an HI/OI ratio <1 (Figure 9A), reflecting the input of material 456 from a terrestrial origin (Carrie et al., 2012). In detail, the values of these two indicators of 457 OM quality reflect an organo-mineral soil horizons origin (A horizon) (Disnar et al., 2003). 458 459 Conversely, the U1 facies of both ponds are characterised by an HI/OI ratio of approximately 2 (Figure 9A), suggesting that the OM origin is either aquatic (Carrie et al., 2012) or, with 460 respect to the HI and OI values, detrital (OH or A horizons) (Disnar et al., 2003). Therefore, 461 OM in the U1 facies is less altered and may have a mix aquatic/terrestrial origin, than that 462 stored in the U2 facies. 463

Spectrocolorimetry data for U2 showed a sedimentary dynamic mainly structured around 464 two end-members which are poles "clayey deposits" and "carbonate deposits" (Figure 9B). 465 The clayey deposits pole reflects the input of detrital clay sediments while the carbonate 466 deposits pole reflects the input of light sediments because of their carbonate contents. These 467 results were confirmed by FDS and show a predominance of the carbonate signature (Debret 468 et al., 2011). The U2 facies is therefore the consequence of the sedimentation of a clay-469 carbonate material, with an organic component that had a terrestrial origin. This terrigeneous 470 deposit, which accumulated in a low intensity zone, may be derived from the erosion of the 471 carbonate substratum of the Seine River watershed. 472

In terms of the U1 facies of both ponds, there was sedimentation consisting of fine sediments enriched in OM. Indeed, the U1 facies is close to the poles of "clayey deposits" and "organic-rich sediment (*Chlorophyll-a and by-products*)" (Figure 9B). This organic pole, also confirmed by the FDS (Debret et al., 2011), could be explained by high aquatic primary production and also by a less degraded soil OM. Such mixing is also verified by the pseudo Van-Krevelen diagram.

479 The similar trend of the U1 facies from the two ponds, and the absence of a clear depth distribution in the pseudo Van-Krevelen and Q7/4 diagrams (Figure 9), suggest that (i) SPM 480 are not altered by any processes between the ~ 10 km separating the two ponds and, (ii) 481 sediment deposits were not submitted to post-depositional alterations (e.g., early diagenesis), 482 probably due to a high sedimentation rate (Van Metre et al., 1997; Horowitz et al., 1988; 483 Callender and Robbins, 1993; Callender, 2000; Audry et al., 2004). Hence, there is a common 484 origin of sediments from both U1 facies, corresponding to the inputs from the Eure River (U1 485 = Eure Unit) and consequently, intrusions of the Seine River into Martot Pond (during tidal 486 flow) do not alter the Eure River signature. Therefore, the U1 facies from Martot Pond was 487 considered as representative of the Eure River signal recorded in sediments, and so was used 488 489 to build an age model.

490

491 4.3. Links with the morphological modification of the Seine and Eure Rivers

492

According to the age model established for the MAR15-01 core, the transition between the U2 facies and the Eure Unit date from around 1954 ± 10 y. The history of the study site showed that Martot Pond was disconnected from the Seine River from 1939. The recently shaped connection between Martot Pond and the Eure River seems consistent with this change, or else dated from the early 1940s. The age model therefore seems consistent with the morphological modification, and the facies change was established at 1939–1944, when Martot Pond was isolated from the Seine River.

The U2 facies was deposited before this major change. The history of the study site shows that, before the Eure River oulet was diverted, the present Martot Pond corresponded to the area behind the Martot Dam, supplied by waters from the Seine River. The U2 facies corresponds to sedimentation of material from the Seine River watershed upstream of the dam. Moreover, the GPR prospecting showed that, in some places, the potential lower limit of this unit corresponded to parts of the island seperating the two branches of the Seine River.

The sediment stored in Martot Pond would be useful to study the Eure River basin from the early 1940s. However, the lack of dating means that, to date, the U2 facies (Seine Unit) has not been analysed in detail and will be the subject of a future prospsection.

509

510 4.4. Identification of an anthropogenic imprint in the Eure River facies

511

According to the correlation matrix, the major elements (Si, Fe, Al, and Ti) were strongly correlated with each other (0.69 < R < 0.87) (Figure S1). The input of these major elements represents the detrital inputs from the Eure River watershed. The trace elements (Zn, Cu, Ni, and Pb) were not correlated with major elements, demonstrating that their presence within the

Eure Unit was not linked to the natural input of the Eure River watershed. In addition, Zn, Cu, 516 and Ni were positively correlated with each other (0.79 < R < 0.88), indicating a common 517 source in the watershed (Figure S1). The lack of correlation between TME and fine particles, 518 as well as the homogeneity of the grain size distribution, indicates that the TME variations 519 520 were related to anthropogenic contributions rather than lithological variations. This lack is also seen with the OM, because the variation in TOC vs TME is not correlated. Lastly, the 521 sedimentation rate does not control the TME variations, as the latter was estimated as constant 522 523 over the considered period.

As a consequence, the temporal variations in the semi-quantitative Zn, Cu, Ni, and Pb data were related to changes in the input of particulate TME. These variations may reflect the presence of new sources within the watershed, and may be related to anthropogenic activities that occurred during the second half of the twentieth century.

528

529 4.5. Reconstruction of Pb history

530

In large European watersheds, Pb levels generally exhibit similar temporal trends with 531 increases from the 1940s until the 1970s, then decreases until the 1990s (Danube and Rhine 532 Rivers : Winkels et al., 1998; Loire River : Grosbois et al., 2006; Seine River : Le Cloarec et 533 al., 2011; Ayrault et al., 2012; Rhône River : Ferrand et al., 2012). Conversely, in the Eure 534 River, Pb levels were stable during the 1960s, increased at the end of the 1980s, and reached a 535 maximum in the 1990s and 2000s followed by a decrease after 2006 (Figure 10). The high 536 537 Pb/Ti ratio values in Martot Pond in the 1990s and 2000s reflect the high Pb concentrations measured along the MAR16-02 core. This high concentration was also seen in SPM and 538 dissolved load collected in the lower reaches of Eure River from March and September 2009 539 (Chiffoleau et al., 2012). In detail, the concentrations were 4-5 times (for particulate 540 concentrations: $Pb_p = 430$ and 380 mg kg⁻¹; measured on SPM) and 5–10 times (for dissolved 541

concentrations: $Pb_d = 1400 \text{ ng } L^{-1}$ and 800 ng L^{-1}) higher than those measured in the Seine 542 River at Poses and during the same period. Chiffoleau et al. (2012) hypothesised that the high 543 Pb levels were not related to atmospheric input but rather to a cathode-ray tube (CRT) factory 544 containing 1 to 3 kg of Pb (Tsydenova and Bengtsson, 2011) in their leaded glass (Singh et 545 546 al., 2016). The CRT factory started operating at Dreux in 1956 and a second factory, specialising in the assembly of CRT TVs and the manufacture of electrical circuits, opened in 547 1974 in the same industrial site. The Pb content in the sediments showed little evidence of the 548 549 successive commencement of these two factories (Figure 10). The industrial production increased from 1990 to the 2000s, when the factories were taken over by the Philips Group, 550 increasing the Pb discharge from the site. To illustrate our statement, the European 551 552 Environment Agency reported a direct release of 3540 kg of Pb into the water in 2001, exposing it as one of the highest European Pb emitters. Between 2002 and 2005, production 553 decreased, as did Pb release (1900 to 1500 kg of Pb into the water in the years 2003 and 2004) 554 ("IREP," n.d.). At the commencement of industrial growth, the Pb/Ti ratio began to increase, 555 and reflecting the instantaneous reactivity of the watershed to this anthropogenic disturbance. 556 However, the fall in industrial output did not produce a decrease in the Pb/Ti ratio, which 557 continued to rise into the late 2000s when the industrial sites were definitively closed (Figure 558 10). Again, this signifies that the watershed reacts to anthropogenic changes, however, the 559 decrease in the ratio is slower than initially expected and remains high after the termination of 560 industrial activity. As an example, near the top of the core, the Pb concentration is twice as 561 high as than the average concentration estimated before 1990. As the levels of the Pb/Ti ratio 562 and the Pb concentrations before industrial activity increased (1990s) may correspond to 563 a reference level of Pb concentration in the Eure River, an additional Pb source is expected in 564 the Eure River watershed. Without any other anthropogenic Pb emitters, it is reasonable to 565 conjecture that this is likely related to Pb contaminated soil surrounding the factory and Pb 566 contaminated sediments stored in the riverbanks and channel, which fed Pb into the river 567

during erosion processes. Hence, the watershed was not able to react immediately to the cease of industrial activity, and a resilience period of the system could be identified (Holling, 1973; Folke et al., 2002). In this case, and due to the erosion of contaminated soil and sediments further realising Pb into the environment, it will be take several years or decades for the river to reach the reference level in Pb concentration. The close relationship between this industrial activity and the observed temporal trends in Pb content suggests that this activity is the main source of contamination but does not allow to state that it is the only one.

575

576 4.6. Reconstruction of Zn, Cu, and Ni

577

578 First, the sharp increase in Zn, Cu, and Ni at the transition between the Seine and the Eure Units shows how the modification of the Seine River channel (for navigation) instantly 579 impacted the quality of the sediments deposited in Martot Pond. The historical trends of Zn, 580 Cu, and Ni, reported by several studies in large European rivers, such as the Danube, Rhine, 581 Loire and Seine basins, show that the concentrations increased between 1940 and the 1970s, 582 then decreased (Winkels et al., 1998; Grosbois et al., 2006; Le Cloarec et al., 2011). Just as in 583 these watersheds, the Zn, Cu, and Ni contents in the Eure River watershed were high until the 584 1980s and peaked during the 1960s and 1970s (Figure 10). The strong correlations between 585 these elements suggest a single source to the Eure River, that started before the facies 586 transition, with the Wonder battery factory the likely source. This factory near the Eure River 587 (20 km upstream from Les Damps) manufactured saline batteries (from Zn) and supplied 588 nearly 40 % of the national market in 1966. The history of this industrial activity indicates 589 that in 1971, the factory was the subject of an increased administrative survey by a national 590 agency, prompting reduced discharge into the Eure River (Agence Financière de Bassin 591 "Seine-Normandie," 1971). This survey is recorded in the sedimentary archive and 592 corresponds to the beginning of the decrease in Cu (Figure 10). Due to a global evolution of 593

the industry (relocation, change in type of batteries), this industry declined in the late 1960s, 594 which is reflected in the Zn, Cu, and Ni contents in the sediments. After the termination of 595 industrial activity (1994), the Zn, Cu, and Ni content remains stable. Here, the watershed 596 seemed to react instantly with the change in anthropogenic activity. Even if we had no 597 598 historical and industrial arguments to state that this industrial site was the single source of TME in the river, we would suggest that it was the main one, which is supported by the close 599 600 relationship between its activity and the observed trends in Zn, Cu, and Ni content in the studied core. 601

The stability of the ratios may indicate that TME release has ceased, but this has to be monitored as there is no record of the TME concentrations before the factory opened in 1938 (Guermond, 1981). Hence, it is therefore impossible to confirm whether the precontamination levels have been restored or whether contaminants are still being released, even at lower levels, as was the case for Pb.

607

608 5. Conclusion

609

The modification of the Seine River morphology since the nineteenth century has generated depositional zones in the lower Eure River. The transition between the Seine River and the Eure River units was the marker of one of the last main morphological changes undertaken in the Seine River Estuary.

A geophysical investigation of both ponds showed a homogeneous filling validating the representativeness of the studied sediment cores. Consequently, our results revealed a regional signature and are not linked to some local conditions.

617 The sedimentary dynamic during deposition and the nature of the sediment were determined618 to ensure that the TME evolution over time was related to anthropogenic activities.

The study of legacy sediments allowed the reconstruction of the industrial trajectory of the watershed since the 1940s. Our results suggested that the Eure River watershed was submitted to high human pressures that could have affected the Seine Estuary.

We have demonstrated that the watershed is very sensitive to any disturbance though a resilience period occurs that extends beyond the period of these disturbances, due to erosion of contaminated soil and sediment.

In the course of the Anthropocene, sedimentation zones containing contaminated sediments may be impacted by extreme events (e.g. floods) or modified by human activities (e.g. dam destruction, dredging, and channelisation), which may cause a resuspension of these sediments within rivers, constituting a new source of contamination. Such complex processes concern most worldwide rivers, and for the foreseeable future, it will become necessary to determine their own resilience periods.

631

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637

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Figure 1. A. Historic sediment core sites in the lower reaches of the Seine River watershed and sediment core sites in the Eure River (present study). The length, time period represented and chemicals elements studied for each core sites are also mentioned; B. The Eure River watershed (present study). The Yonne and Oise rivers sub-watersheds studied in Ayrault et al., 2010b and Le Cloarec et al., 2011 are also represented.

Figure 2. Morphological modifications of Martot Pond and Les Damps Pond study sites established from several unpublished documents (e.g. maps from "Pont et Chaussées" and meeting minutes) stored at "Voie Navigable de France" (VNF). The cities of Martot, Pont-de-l'Arche et Les Damps have been indicated on the maps to facilitate comparison with the actual study area.

Figure 3. A. Study Area; B. Sediment core and GPR profiles locations in Les Damps Pond; C. Sediment core and GPR profiles locations in Martot Pond.

Figure 4. A1. Colour log; and B. Grain size distribution and D₅₀; A2. Colour log; C. TOC, HI, and OI; D. Magnetic susceptibility (MS) and reflectance (L*); E. First derivative values; F. Pb/Ti, Zn/Ti, Cu/Ti, and Ni/Ti ratios along the DAM15-02 and DAM17-02 cores collected in Les Damps Pond.

Figure 5. A. Colour log; B. Grain size distribution and D50; C. TOC, HI, and OI; D. Magnetic susceptibility (MS) and reflectance (L*); E. First derivative values; F. Pb/Ti, Zn/Ti, Cu/Ti and Ni/Ti ratios along MAR15-01 core collected in Martot Pond.

Figure 6. Pb/Ti, Zn/Ti, and Cu/Ti ratios along the upper part of the MAR16-02 core. Pb (dark circle), Zn (dark square) and Cu (dark triangle) concentrations are also presented.

Figure 7. A. ²¹⁰Pbex; B. ¹³⁷Cs and. ²⁴¹Am; C. Age model for the MAR15-01 core.

Figure 8. Radargram performed with a 400 mHz antenna, migration rate used: 0.075 m ns⁻¹. Due to the lengths and depths reached and to obtain adequate readability, the x-scales of the 3 profiles are different. For reasons of navigability, all the core sampling points previously presented could not be documented by GPR, in particular the MAR15-01 and DAM15-02 cores A. GPR-MAR_08 radargram, processed and interpreted from Martot Pond. To obtain a 2D information, the MAR15-02, MAR15-03, MAR15-04, and MAR15-06 cores with a simple description distinguishing only the U1 and U2 facies, have been positioned; B. GPR_DAM_03 radargram processed and interpreted from Les Damps Pond. The DAM15-03 and DAM18-02 cores have been positioned; C. GRP_DAM_05 radargram, processed and interpreted from Les Damps Pond. The DAM17-02 core has been positioned.

Figure 9. A. Pseudo Van-Krevelen diagram; B. Q7/4 vs L* ratio for the MAR15-01 core (U1 and U2 facies) and the DAM17-02 core.

Figure 10. Evolution of Pb/Ti, Zn/Ti, Cu/Ti, and Ni/Ti ratios along the Eure Unit (MAR15-01 core).

Figure S11. Correlation matrix with XRF core scanning data (Al, Si, Fe, Ti, Mn, Zn, Cu, Ni, and Pb), TOC, and grain size (D₅₀, clay ($\leq 2 \mu m$), silt (2-63 μm), and coarse fraction (> 63 μm)) from the Eure Unit of the MAR15-01 core (Pearson correlation, p-value ≤ 0.001 for all the R ≤ -0.6 and R > 0.6).

Tableau 1. Characteristics of sediment cores collected in Les Damps Pond and Martot Pond (WGS 84).





















Pond	Core ID	IGSN	Longitude (X)	Latitude (Y)	Core Length (cm)
LES DAMPS	DAM15-02	IEM2C0016	1°10'9.05" E	49°18'16.13" N	80
(DAM)	DAM15-03	IEM2C0017	1°10'11.58" E	49°18'15.83" N	67
	DAM17-02	IEM2C000E	1°10'13.26" E	49°18'15.66" N	90
	DAM18-02	IEM2C001C	1°10'11.14" E	49°18'16.09" N	208
MARTOT	MAR15-01	IEM2C0001	1°03'1.68" E	49°17'49.68" N	138
(MAR)	MAR15-02	IEM2C0002	1°03'12.49" E	49°17'50.63" N	93
	MAR15-03	IEM2C0003	1°03'30.45" E	49°18'1.69" N	68
	MAR15-04	IEM2C0004	1°03'23.34" E	49°17'54.44" N	104
	MAR15-05	IEM2C0005	1°03'2.84" E	49°17'49.69" N	161
	MAR15-06	IEM2C0006	1°03'5.49" E	49°17'49.88" N	96
	MAR16-02	IEM2C0008	1°03'2.60" E	49°17'49.30" N	129